Draft Final Addendum No. 2 to Chemical Data Acquisition Plan

Contract No. DACW 41-90-D-0009

Predesign Investigation Remedial Design Former Nebraska Ordnance Plant Operable Unit 1 Mead, Nebraska

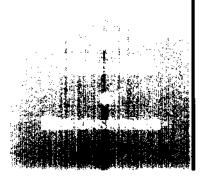
Prepared for:

U.S. Army Corps of Engineers



Department of the Army U.S. Army Engineer District Kansas City Corps of Engineers Kansas City, Missouri

November 7, 1994





DRAFT FINAL CHEMICAL DATA ACQUISITION PLAN

Addendum No. 2 - Predesign Investigation Remedial Design Former Nebraska Ordnance Plant Operable Unit 1 Mead, Nebraska

November 7, 1994

Prepared for:

U.S. Army Corps of Engineers Kansas City District 601 East 12th Street Kansas City, Missouri 64106

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RUST E&I Project No. 72736

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1.0 BACKGROUND AND GENERAL PROJECT APPROACH

Information pertaining to the background of the former Nebraska Ordnance Plant Operable Unit 1 is presented in the Remedial Investigation Report (SEC Donohue, 1992) and Feasibility Study Report (RUST, 1994). Information pertaining to the general project approach is presented in Section 1.0 of the Field Investigation Plan Addendum No. 2.

This second addendum to the Chemical Data Acquisition Plan (CDAP) for the Nebraska Ordnance Plant Remedial Investigation is in accordance with the U.S. Army Corps of Engineers Regulation ER 1110-1-263, October 1, 1990, Engineering and Design, Chemical Data Quality Management for Hazardous Waste Remedial Activities, Appendix D.

2.0 CHEMICAL DATA QUALITY OBJECTIVES

2.1 ANALYTICAL APPROACH

During the former Nebraska Ordnance Plant (NOP) Predesign Investigation, samples to be analyzed for explosives will be collected to further define the horizontal extent of contamination based on preliminary remediation goals (PRGs) calculated by the EPA as part of the feasibility study (RUST, 1994). Samples will be collected to:

- Verify positive field screen data from the RI which exceed remediation goals but did not have laboratory confirmation.
- Further define the source areas previously delineated using the EPA PRGs.
- 3. Conduct field screening analysis for explosives in soil.
- 4. Show that investigation-derived waste (IDW) meets the requirements of the off-site facility where it will be disposed.

Samples will be collected from areas surrounding Load Lines 1, 2, 3, and 4, the Burning/Proving Grounds, and the Bomb Booster Area. The sample locations are presented in Field Investigation Plan Addendum No. 2. Samples will be collected at the specified locations from the 0 to 2-foot and 2 to 4-foot intervals. Table 2-1 presents the anticipated numbers and locations of samples to be collected during the field investigation. Field screening methods are included in Attachment A.

Representative samples of Operable Unit 2 IDW will be collected from 55-gallon drums. One sample of each waste type found within the drums (i.e., sump plastic, personal protective equipment (PPE), and trash) will be analyzed.

2.2 DATA USES

Soil samples will be collected for the analysis of explosives to further define the horizontal extent of contamination based on EPA PRGs. Results from the IDW samples will be used to determine the appropriate means of disposal for the drums containing IDW.

2.3 DATA QUALITY NEEDS

EPA Data Quality Level III was used for explosives analysis during the RI in 1991 and 1992 and the Supplemental RI/FS and Treatability Studies in 1993. To promote the generation of comparable data, EPA Data Quality Level III will be used for explosives analysis for this Predesign Investigation. EPA Data Quality Level III will also be used for the IDW sample analysis. One quality assurance (QA) sample will be required, but no quality control (QC) package will be required from the laboratory and no data validation will be done for the analytical results.

TABLE 2-1

SAMPLING AND ANALYSIS SUMMARY FORMER NEBRASKA ORDNANCE PLANT CDAP ADDENDUM NO. 2 MEAD, NEBRASKA

Sample Source	Sample Matrix	Analysis	Samples to be Field Screened	Samples to Lab	Field Duplicate	Rinsate ⁽¹⁾ Blanks	Total to Lab	QA Splits	Lab QC MS/MSD
Load Line 1	Soil	Explosives	22	22	2	1	25	2	1
Load Line 2	Soil	Explosives	14	14	1	1	16	1	0
Load Line 3	Soil	Explosives	10	10	1	1	12	1	1
Load Line 4	Soil	Explosives	4	4	1	1	6	1	1
Bomb Booster Area	Soil	Explosives	12	12	2	1	15	2	1
Burning/Proving Frounds	Soil	Explosives	32 ⁽²⁾	32(2)	3	1	36	3	2
IDW	Plastic, PPE, &Trash	VOC SVOC Metals Ignitability Corrosivity Reactivity Cyanide Sulfide Paint Filter Total Solids Total Phenolics	0	One per Waste Type		0	One per Waste Type	. 1	1

IOTES:

- QA Splits = The number of Quality Assurance (QA) samples anticipated to be sent to MRD Laboratory.
- Lab QC MS/MSD = The number of laboratory Quality Control (QC) Matrix Spikes and Matrix Spike Duplicate (MS/MSD) pairs anticipated to be analyzed by the subcontract laboratory.
- IDW = Investigation Derived Waste
- PPE = Personal Protective Equipment
- VOC = Volatile Organic Compounds
- SVOC = Semi-Volatile Organic Compounds
- Rinsate blanks will be collected at a frequency of one per day to evaluate the effectiveness of decontamination of field sampling equipment.
- Includes 20 shallow soil samples and 12 samples from former disposal trench test pits.

Field screening methods for explosives will be EPA Data Quality Level I.

EPA Data Quality Levels are defined in <u>Data Quality Objectives for Remedial Response Activities</u>. <u>EPA/540/G-87/003</u> (March 1987).

2.4 PARCC PARAMETERS

Table 2-2 indicates the precision, accuracy, and representativeness criteria for the sample analyses. These data quality parameters are expressed as goals due to uncertainties regarding field conditions and the complexity of the sample matrices at the site. The ability to extract and quantify the target analytes is dependent on the matrix complexity. The precision, accuracy, representativeness, completeness, and comparability parameters are described in the following sections.

2.4.1 Precision

Precision is presented in Table 2-2 as relative percent difference (RPD) between matrix spike and matrix spike duplicate (MS/MSD) samples. RPD of laboratory MS/MSD samples is used to assess the analytical precision of the method. It is assumed that a low RPD represents a high degree of analytical precision. For duplicate results D_1 and D_2 , the RPD is calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{|D_1 + D_2|} \times 200\%$$

2.4.2 Accuracy

Accuracy is presented in Table 2-2 as percent recovery limits for analytical matrix spikes. A known gradient of analyte is added to a sample aliquot and the percent recovery of the known amount of analyte is used to determine the analytical bias (accuracy). Analytical matrix spike results are calculated as follows:

% Recovery =

Concentration in Spiked Sample - Concentration in Unspiked Sample x 100% Concentration of Spike

2.4.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of site conditions. Representativeness will be satisfied by insuring that the appropriate plans are followed, proper sampling technique are used, proper analytical

TABLE 2-2

PARCC PARAMETER GOALS FORMER NEBRASKA ORDNANCE PLANT CDAP ADDENDUM NO. 2 MEAD, NEBRASKA

		Precision	Accuracy		
Analyte Group	Matrix	Matrix Spike Duplicates (RPD)	Matrix Spike (% Recovery)	Surrogate Spike	Trip Blank
Explosives Compounds	Soil and Water	All Compounds: 25%	All Compounds: 75 - 125%	NA	NA

NOTES:

RPD = Relative Percent Difference.

NA = Not Applicable.

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procedure are followed, and holding times of the samples are not exceeded in the laboratory. Representativeness will be assessed by the analysis of field duplicate samples. If the RPDs between the field sample and its corresponding field duplicate are less than the limit presented in Table 2-2, it is assumed the sample is representative.

2.4.4 Completeness

Completeness is defined as the total amount of valid data obtained, divided by the amount of data that would be expected under normal conditions using the Field Investigation Plan. Valid data will be that data which includes, after the data validation process, uncoded data and data with codes of U and J. The goal for total data completeness will be 90 percent. If the total data completeness is less than the goal, there may be insufficient data to meet the data quality objectives.

2.4.5 Comparability

Comparability expresses the confidence with which one data set can be compared with another. The use of standard USEPA analytical methodologies to obtain the planned analytical data are expected to provide comparable data.

3.0 PROJECT ORGANIZATION AND FUNCTIONAL AREA RESPONSIBILITY

3.1 OVERVIEW

The RUST project management organization for the NOP Predesign Field Investigation is designed to provide a line of functional responsibility and authority. The RUST project management organization consists of a management control and independent quality control review structure. Basically, this structure provides:

- Clearly identified lines of communication and coordination.
- · Project budget and schedule monitoring.
- Key technical resources management.
- Periodic financial management and progress reports.
- Quality control.

3.2 RESPONSIBILITIES

In the following sections, the responsibilities of each project team member relative to the overall technical and QC/QA objectives of the project are identified.

3.2.1 Project Responsibility

Ms. Natalae Tillman is USACE's Technical Manager (TM) and will be responsible for coordinating with the RUST Project Manager.

Mr. Richard Fedler, P.E., is Principal-In-Charge for RUST. Mr. Fedler will be responsible for all contractual matters relating to the prime contractor.

Mr. Chandler Taylor is the RUST Project Manager (PM). As PM, he will coordinate the administrative and production portions of the investigation including client billing and processing of subcontractor invoices. The PM will develop an internal project plan, implement the plan, monitor the efforts in relation to the plan, evaluate deviations from the plan, and take appropriate action. The PM will monitor budgets and charges for the entire project. The PM is responsible for developing monthly progress reports with schedule updates as required and submittal of confirmation notices. Mr. Taylor will be the main contact for technical issues for all phases of the project. He will coordinate and direct the technical aspects of the project and coordinate Quality Control (QC) reviews. Mr. Taylor is responsible for all technical activities and schedule.

The Field Team Leader (FTL) is Mr. Matt Stebbins. He will report to the PM and coordinate and be responsible for pre-field planning and field activities. Prefield activities include preparing subcontract or specifications and subcontract for signature by the RUST Division Manager, ordering field equipment, and conducting a pre-field briefing meeting. Additional detail on the FTL responsibilities is discussed in Section 2.1 of the FIP.

The field team consists of personnel from RUST and from environmental subcontractor(s). The field team report(s) directly to the FTL. Additional data on the responsibilities of the field team is discussed in Section 2.2 of the FIP.

Mr. Richard Tinsley is the Regional Health and Safety Specialist (RHSS). The RHSS will implement the Site Health and Safety Plan (HASP) for the project through an assigned Site Safety Officer (SSO) and will enforce site safety rules. The RHSS will review immediate site problems encountered directly with the PM.

The Laboratory/Data Coordinator is responsible for the coordination between RUST and the analytical laboratory as well as assessing the quality of analytical data generated.

3.3 ASSIGNED INDIVIDUALS

The following is a listing of the key personnel assigned to this project and their area of responsibility.

Natalae Tillman Richard Fedler Chandler Taylor Matt Stebbins Richard Tinsley B.J. LeRoy Steve Grumann USACE Technical Manager
Principal-In-Charge
Project Manager
Field Manager
Regional Health and Safety Specialist
Site Health and Safety Officer
Field Laboratory/Data Coordinator

3.4 LABORATORY RESPONSIBILITY

The subcontract and QA laboratories which have been selected for this project are:

Huntingdon/TCT-St. Louis
 1908 Innerbelt Business Center Drive

 St. Louis, Missouri 63114

 (314) 426-0880

 (314) 426-4212

Point of Contact: Paul Smith

USACE MRD QA Laboratory
 420 S. 18th Street
 Omaha, NE 68102
 (402) 444-4313
 (402) 341-5448 (Fax)

Point of Contact: Laura Percifeld QA Analyses

A copy of the USACE MRD letter of validation is included as Attachment B to this CDAP Addendum.

4.0 SAMPLING PROGRAM

Specific sampling procedures are described in the Field Investigation Plan Addendum No. 2.

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5.0 SAMPLE CHAIN OF CUSTODY, PACKAGING, AND SHIPPING

5.1 SAMPLE CUSTODY

It is RUST E&I policy to follow the U.S. EPA chain of custody protocols as described in <u>NEIC Policies and Procedures</u>, EPA 330/9-78-001-R, Revised June 1985. This custody is in three parts: sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports, are maintained under document control in a secure area.

A sample or evidence file is under custody if it:

- is in the possession of the sampler/analyst.
- is in the view, after being in the possession of the sampler/analyst.
- is in the possession of the sampler/analyst and then placed in a secured location.
- is in a designated secure area.

5.1.1 Field Custody Procedures

The goal of the sample handling procedures summarized below is to ensure that the samples will arrive at the subcontract and QA laboratories with the chain of custody intact.

5.1.1.1 Field Data Coordinator to Field Sampler

The Field Data Coordinator (FDC) is responsible for initiating the chain of custody of the sample containers. As few people as possible will handle the samples.

The FDC will label each container indicating the sample location and the preservative required, if any, for that container. Sample numbering protocol is discussed in Section 4 of the FIP Addendum No. 2. The FDC will initiate the chain of custody by recording the following information on a Field Custody Card (Figure 5-1):

- Sample location.
- Summary of the number and type of sample containers, for example: 1 8 oz. glass jar.

The FDC will transfer custody of the containers to the field sampler by signing the first line on the back of the Field Custody Card and recording the date and time. The field sampler will verify that the sample containers being transferred are properly recorded on the card, and will accept custody of the containers by signing the second line on the back of the Field Custody Card.

5.1.1.2 Field Sampler to Field Data Coordinator

The field sampler will collect the samples and will store them in a cooler with ice. The field sampler will complete the following information on the Field Custody Card:

FIGURE 5-1 FIELD CUSTODY CARD

Former Nebraska Ordnance Plant CDAP Addendum No. 2 Mead, Nebraska

Sample Number	Number of Bottles and Type	Depth	PID	Date	Time
		:			
					;
		į			
*PE (Presumpress					
		i	į l	ı ,	

Front

Refinquished By	Date To	Time	
Relinquished By	Oste	Time	
	То		
			

Back

- Depth that the sample was collected from, if applicable.
- PID reading of the sample, if applicable.
- · Date and time of sample collection.

The cooler in which the samples are stored will remain in sight of the sampler or in a locked area, such as a vehicle or job trailer. The field sampler will transfer custody of the samples to the FDC by signing the third line on the back of the Field Custody Card and recording the date and time. The FDC will verify that the samples being transferred are properly recorded on the card and will accept custody of the samples by signing the fourth line on the back of the Field Custody Card.

The information provided on the Field Custody Card will be used to complete the sample labels (Figure 5-2), Chain of Custody Record (Figure 5-3), and the Field Sample Log. The FDC will affix the appropriate sample label to the container. The completed Field Custody Cards will be stored on-site with other sample documentation. The Field Sample Log will be completed by the FDC. Information to be entered into the Field Sample Log include:

- Sample label number.
- Field sample identification number.
- Sample matrix.
- Date and time sample was collected.
- Initials of field sampler
- Analyte(s).
- Type of sample container.
- Preservative, if any.
- Whether the sample is a field duplicate, rinsate blank, or trip blank.
- Laboratory destination.
- Date of sample shipment.
- Airbill number.

5.1.1.3 Field Data Coordinator to Laboratory

Samples in the possession of the FDC which have not been packaged for shipping will be kept in a secured area. Bags of ice will be packed around and on top of the samples to maintain the temperature of the samples.

Samples will be properly packaged for shipment (Section 5.2) and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample shipping container. The shipping container will be latched and secured with strapping tape. The FDC will initial and date the custody seals (Figure 5-4). Custody seals will be attached to the front right and back left of the shipping container. The custody seals will be covered with clear plastic tape. The shipping container will be sealed with strapping tape in at least two locations.

A Chain of Custody Record will be initiated by the FDC. The label numbers, sample identification numbers, date and time sample was collected, and FDC's signature will be listed on the Chain of Custody Record. When transferring the possession of samples, the individuals relinquishing and

FIGURE 5-2 SAMPLE LABEL Former Nebraska Ordnance Plant CDAP Addendum No. 2 Mead, Nebraska

	vironment & Infrastructure rth 40th St., Sheboygan, WI 53082
Client:	
Site: _	
Sample I	No: Sample Name
•	to: Sample Name 5: Analyte Mame
Analysi:	•

FIGURE 5-3 CHAIN OF CUSTODY FORM Former Nebraska Ordnance Plant CDAP Addendum No. 2 Mead, Nebraska

Sample Type Sun ple Type Sun pl
--

FIGURE 5-4 CUSTODY SEAL Former Nebraska Ordnance Plant CDAP Addendum No. 2 Mead, Nebraska

RUST ENVIRONMENT & INFRASTRUCTURE

Custody Seal #

receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the FDC to the laboratory, or to/from a secure storage area on-site.

Shipments will be accompanied by the Chain of Custody Record identifying the contents. Samples being shipped to USACE MRD Lab will have MRD project Laboratory Information Management System (LIMS) number recorded on the Chain of Custody Record. The original copy (white) and yellow copy will accompany the shipment, and the pink copy will be retained by the sample custodian for return to the project files which are kept under the custody of the RUST E&I Project Manager.

If the samples are sent by common carrier, a bill of lading will be used. Receipts of bills of lading will be retained as part of the permanent documentation. Commercial carriers are not required to sign off on the custody forms as long as the custody forms are sealed inside the shipping container and the custody seals remain intact.

5.1.2 Laboratory Custody Procedures

The following provisions for maintaining custody of samples will be followed by the laboratories performing chemical analysis:

- Access to the laboratory is through a monitored reception area. Other access doors to the laboratory are kept locked.
- Visitors such as clients, regulatory agencies, or the public must sign in at the reception area and be escorted while in the laboratory.
- Samples are stored in a secure area. Only the sample custodian has access; analyst must obtain samples through the sample custodian.
- Custody records are maintained by the sample custodian.
- After a sample has been removed from storage by the analyst, the analyst is responsible for the
 custody of the sample. Each analyst must return the samples to the storage area before the end
 of the working day.

The laboratory sample custodian either accepts or rejects custody of the sample. This is based on:

- Compliance with holding times.
- · Proper/incorrect sample preservation.
- Presence/absence of custody seals on the cooler.
- Condition of cooler.
- Presence/absence of Chain of Custody Record.
- Presence/absence of air bills or other bills of lading.
- Condition of samples (intact, broken, leak, etc.).
- Temperature of sample (4°C ±2).
- Presence/absence of sample labels.

Agreement between labels and Chain of Custody Record.

This information is documented on a Cooler Receipt Form (Figure 5-5).

If custody of the sample is rejected, or the integrity of the sample is questioned, the RUST E&I QC Officer is advised as soon as possible. If custody of the sample is accepted, the laboratory sample custodian will sign, date, and note the time on the Chain of Custody Record. The sample is then logged into the laboratory system. A new unique laboratory sample number will be assigned to each sample to keep the analyst blind to any laboratory introduced QC samples, field blanks, and field duplicates. All participating laboratories have laboratory sample tracking programs with secure restricted access to data.

5.2 PACKAGING AND SHIPPING

5.2.1 Sample Packaging

Samples will be shipped according to the following instructions:

- 1. Attach completed adhesive sample label to the sample container.
- 2. Secure container lids with strapping tape (except for VOC samples).
- 3. Place container in appropriate size Ziploc bag and seal.
- 4. Wrap bagged glass container in bubble wrap or foam sleeve to minimize breakage.
- 5. Select an appropriately sized metal or plastic cooler (shipping container) and tape the drain plugs on the inside and outside with duct tape.
- 6. A label will be placed on top of the shipping container and will include sample shipper's name, mailing address, and phone number; the laboratory's name, mailing address, phone number, sample quantities, date of shipment, and indication that samples are "environmental samples" (Figure 5-6). The appropriate sides of the container will be marked "This End Up" and arrows attached accordingly. "Fragile" labels will be placed on the cooler lid.
- 7. Line the cooler with a large polyethylene garbage bag.
- 8. Put absorbent packaging material on the bottom of the polyethylene garbage bag and put samples in the cooler with bags of ice around and over the samples. Fill the cooler with the absorbent packaging material to reduce the possibility of breakage.
- 9. Place a completed Chain of Custody Record (white and yellow copies) listing the contents of the cooler, a Cooler Receipt Form, and a completed cooler return address/label in a Ziploc bag and tape to the underside of the cooler lid.

FIGURE 5-5 COOLER RECEIPT FORM

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	MRD Cooler No.;		
Pro	ect Date Received;		
eg	other side of this form to note further details concerning check-in problems and to specify and describe urding the resolution(s) of problems. If shipment was accepted and if requested note on back the address ty cooler was returned and likewase if the shipment was rejected.	any acus S where t	on(s) the
_	PRELIMINARY EXAMINATION PHASE: Date cooler was opened:		
	by (print) (sign)		
	Were custody seals on outside of cooler? If YES, how many and where?	YES	NO
	Date and signature correct? If YES, seni date: Name:	YES	NO
	2. Were custody seals unbroken and intact at the date and time of arrival?	YES	NO
	3. Were custody papers sealed in a plastic bag and taped inside to the lid?	YES	МО
	4. Was project identifiable from custody papers? If YES, enter project name at top of this form	YES	Ю
	5. Were custody papers filled out property (ink, signed, etc.)?	YES	NQ
	6. Did you sign papers in the appropriate place?	YES	NO
	7. Did cooler come with a shipping stip (air bill, etc.)? If YES, attach and enter air bill or invoice number here:	YES	NO
	8. Have designated person initial here to acknowledge receipt of cooler: (date)	•	
3.	LOG-IN PHASE: Due samples were logged in:		
	by (print) (sign)		
	9. Describe packing:	· - ·	
	10. If required, was enough see used? (Temperature =)		Ю
	11. Were all bodies sealed in separate plastic bags?	YES	NO
	12. Did all bottles arrive unbroken and in good condition?	YES	NO
	13. Were all bottle labels complete (ID, date, time, signature, preservative, etc.)?	YES	NO
	14. Did all bottle tabels agree with custody papers? If NO, indicate discrepancies on back.	YES	NO
	15. Were convect consumers used for the tests indicated?	YES	МО
	16. Were correct preservatives used when required?	AE2	NO
	17. Was a sufficient amount of sample sent for tests indicated?	YES	МО
	18. Bubbles abesent in VOA vials? If NO, list by QA No.:	YEŞ	NO

FIGURE 5-6 SAMPLE SHIPPING LABEL Former Nebraska Ordnance Plant CDAP Addendum No. 2 Mead, Nebraska

April 28, 1994

Sample Custodian
RUST Environment and Infrastructure
4738 N. 40th Street
Sheboygan, WI 53083
(414) 458-8711

To:
Analytical Laboratory
Address
City, State Zip Code
Telephone Number

Contents:
Environmental Samples
6-1L Glass Bottles, 2-1L HDPE Bottles, 12-40ml Vials

- 10. Close the cooler and seal with strapping tape around it in at least two places.
- 11. Place the signed and dated Custody Seal on the cooler at the left back and front right of cooler so if the cooler was opened, the seals would break. Cover the seals with clear tape.
- 12. Complete and attach the courier air bill.

5.2.2 Shipment Coordination

Before field work begins, the laboratories will be notified of a preliminary sample shipment schedule. The FDC will call the RUST E&I laboratory coordinator or fax the Chain of Custody Forms. The RUST E&I laboratory coordinator will notify the laboratories performing chemical analyses and the USACE-MRD Lab of sample shipments on the day of shipment. At that time, the field sample coordinator will provide the following information:

- 1. Site name.
- Number(s) and matrix(ces) of samples.
- Carrier name.
- Method of shipment (i.e., overnight, two-day).
- 5. Date of shipment.
- Suspected hazards associated with the samples or site, if any.
- Irregularities or anticipated problems with the samples, including special handling instruction or deviations from established sampling procedures or numbers of samples, if any.
- 8. Status of the sampling project (i.e., final shipment, update of future shipping schedule if a change has occurred).

5.3 FINAL EVIDENCE FILES CUSTODY PROCEDURES

The final laboratory evidence files from the laboratory will be assembled by the RUST E&I QC Officer and relinquished to the Project Manager.

RUST E&I will maintain the laboratory files along with relevant records, reports, logs, field notebooks, pictures, subcontractor reports, and data reviews in a secured, limited-access area, and under custody of the RUST Project Manager.

6.0 LABORATORY ANALYTICAL PROCEDURES

6.1 INTRODUCTION

This project will include analyses of soil samples and rinsate blanks for explosives by EPA's Method 8330 from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition. Detection limits for explosives and field screening techniques are defined in Table 6-1. Internal quality control check limits are defined in Table 6-2. Descriptions of the internal quality control checks are described in the CDAP Addendum No. 1.

IDW waste will also be analyzed for the parameters shown in Table 6-3 using SW-846 methods. Detection limits will be set such that results can be compared to the regulatory levels shown on the Douglas County Landfill Special Waste Analytical Requirements Form included in Attachment C.

6.2 EXPLOSIVES

Soil and rinsate samples will be analyzed for explosives following SW-846 Method 8330, as outlined in CDAP Addendum No. 1.

Soil samples being field screened will be analyzed for explosives following the method(s) presented in Attachment A.

TABLE 6-1

EXPLOSIVES DETECTION LIMITS FORMER NEBRASKA ORDNANCE PLANT CDAP ADDENDUM NO. 2 MEAD, NEBRASKA

Explosive Compound	Laboratory Soil Sample (mg/kg)*	Laboratory Water Sample (ug/l)	Soil Field Screen (mg/kg)
HMX	2.2	13.0	
RDX	1.0	14.0	0.5
1,3,5-Trinitrobenzene	0.25	7.3	
1,3-Dinitrobenzene	0,25	4.0	
Tetryl	0.65	44.0	
Nitrobenzene	0,26	10.0	
2,4,6-Trinitrotoluene	0,25	6.9	0.25
2,4-Dinitrotoluene	0.25	5.7	
2,6-Dinitrotoluene	0.26	9.4	
o-Nitrotoluene	0.25	12.0	
m-Nitrotoluene	0.25	7.9	
p-Nitrotoluene	0.25	8.5	
2-Amino-4,6-dinitrotoluene	0.25	5.0	
4-Amino-2,6-dinitrotoluene	0.25	5.0	

NOTES:

*Detection limits are matrix dependent and may actually vary from those listed.

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TABLE 6-2

FREQUENCY OF INTERNAL QUALITY CONTROL CHECKS FORMER NEBRASKA ORDNANCE PLANT CDAP ADDENDUM NO. 2 MEAD, NEBRASKA

Parameter	Method Blank	Matrix Spike	Matrix Spike Duplicate	Known Reference	Surrogate Spike	Trip Blank
Explosives	One Per Batch Extracted	1/20	1/20	1/20	NA	NA
NOTES:	- ,, ,					
NA = Not Ap	plicable.					

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TABLE 6-3

INVESTIGATION DERIVED WASTE ANALYTICAL PARAMETERS AND METHODS FORMER NEBRASKA ORDNANCE PLANT CDAP ADDENDUM NO. 2 MEAD, NEBRASKA

Parameter	Method ⁽¹⁾
voc	8260
SVOC	3550/8270
Metals ⁽²⁾	3050/6010, 7061, 7740, 7471A
Ignitability	1020
Corrosivity	9045
Reactivity	Section 7.3, Chapter 7
Cyanide	9012
Sulfide	9030
Paint Filter	9095
Total Solids	EPA 160.3 ⁽³⁾
Total Phenolics	9066

NOTES:

VOC = Volatile Organic Compounds

SVOC = Semi-Volatile Organic Compounds

- SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, EPA, September 1986, unless specified otherwise.
- (2) Includes (8) RCRA Metals Plus Copper, Nickel, and Zinc.
- (3) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020.

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7.0 CHEMICAL DATA QUALITY MANAGEMENT DELIVERABLES

7.1 QUALITY CONTROL/QUALITY ASSURANCE SAMPLES

Quality control reports will be completed daily for field activities using the Daily Quality Control Summary Report (DQCR) forms. The DQCR will include information about the field activity, including the date, current weather conditions, persons present on-site, project name and number, equipment on-site, description of work being performed, reporting of any quality control activities, personal protective equipment being used, any problems or corrective actions noted, and the signature of the person preparing the DQCR.

7.1.1 Quality Assurance Samples

For every 10 samples collected, or less, one sample will be collected in triplicate to assess the quality of the sampling and the analytical laboratory procedures. Two of the samples will be sent to the analytical laboratory and labelled so as not to identify them as the duplicates. The duplicates will assess the precision of the sampling procedures. The third sample will be analyzed by the USACE Missouri River Division Laboratory (MRD) in Omaha, Nebraska, to assess the accuracy of the analytical laboratory as well as the precision of the sampling procedures. Methods for collection of these samples are described in the Field Investigation Plan.

7.1.2 Matrix Spikes and Matrix Spike Duplicates

For every 20 samples analyzed by the laboratory, at least one sample will be analyzed along with a matrix spike and matrix spike duplicate. Percent recoveries of the spike and relative percent differences of the spike duplicates will be calculated and reported. If percent recoveries or RPDs are outside of the control limits, it will be noted in the narrative of the report deliverables.

7.2 DATA REPORT TO THE QA LABORATORY

Analytical data generated will be submitted to the RUST Laboratory/Data Coordinator for review. Analytical laboratory data will be reviewed to ensure project QC requirements were met. Data validation will be performed by conducting a systematic review of the data for compliance to the established QC criteria based on the QC results provided by the laboratory. An evaluation of data accuracy, precision, sensitivity, and completeness, based on criteria as discussed in Section 2.0, will be performed and presented in the report.

Data validation will be performed based on procedures outlined in the "National Functional Guidelines for Organic Data Review," June 1991. Qualifiers used during the validation process will indicate that the data are: 1) usable as a quantitative concentration; 2) usable with caution as an estimated concentration [coded J]; or 3) unusable due to out-of-control QC results [coded R].

This data will be retained by RUST for a period of 5 years. This data will in turn be submitted to the USACE QA laboratory along with the following:

- A table which matches the analytical laboratory's sample identification numbers to the QA laboratory sample numbers. This table will identify field duplicates, and field blanks. The table will indicate which field samples are associated with the field blanks and duplicates.
- Completed "Cooler Receipt Forms" (Figure 5-5) for all shipments which will note problems in sample packaging, chain of custody, and sample preservation.
- General Reporting For each analytical method run, the report will indicate analytes for each
 sample as a detected concentration or as less than the specific limits of quantitation. Generally,
 samples with out-of-control spike recoveries being attributed to matrix interferences will be
 designated as such. Soil samples will be reported on a dry-weight basis with percent moisture
 also reported. The report will include dilution factors for each sample as well as the date of
 extraction (if applicable) and date of analysis.
- Internal Quality Control Reporting as submitted by the laboratory (at a minimum, internal quality control samples will be analyzed at rates specified in the specific methods):
 - (1) Laboratory Blanks (Method Blanks and Instrument Blanks) All analytes shall be reported for each laboratory blank. All non-blank sample results will be designated as corresponding to a particular laboratory blank in terms of analytical batch processing.
 - (2) Matrix Spike Samples Matrix Spike Recoveries will be reported for all organic and inorganic analyses. All general sample results will be designated as corresponding to a particular matrix spike sample. The report will indicate what field sample was spiked even if it was not a project sample. The report will also specify the control limits for matrix spike results for each method for each matrix
 - (3) Matrix Spike Duplicate Pairs RPD will be reported for all duplicate pairs as well as analyte/matrix specific control limits.
 - (4) When run for internal quality control, Laboratory Control Standards (LCS) results will be reported with the corresponding field sample data. Control limits for LCSs will also be specified.
- Field Duplicates These samples will be identified as such by RUST and reported as any other field sample. Relative Percent Differences will be reported for all field duplicate pairs.
- Quality Control Summary Report (QCSR) RUST will prepare a draft final QCSR and a revised final QCSR which will include a summary of all Daily Quality Control Reports (DQCR) completed during field sampling. The QCSR evaluates the quality of data and field activities as it relates to the quality control results. The QCSR will be due within 60 days of the availability of analytical results.

7.3 CHEMICAL QUALITY ASSURANCE REPORT

The USACE Kansas City District Project Chemist will review the MRD Laboratory Chemical Quality Assurance Report and, where necessary, RUST will address in writing major discrepancies identified. The Chemical Quality Assurance Report will be included as part of the RI Report.

8.0 REFERENCES

- RUST Environment & Infrastructure (RUST), 1994. Draft Final Feasibility Study, Former Nebraska Ordnance Plant, Operable Unit 1, Mead, Nebraska.
- SEC Donohue, 1992. Supplemental Remedial Investigation Report, Former Nebraska Ordnance Plant, Operable Unit 1, Mead Nebraska.
- U.S. Army Corps of Engineers (USACE), 1990. Chemical Data Quality Management for Hazardous Waste Remedial Activities, CEMP-RT Engineering Regulation No. 1110-1-263, October 1, 1990.
- U.S. Environmental Protection Agency (USEPA), 1987. Data Quality Objectives for Remedial Response Activities, EPA 540/G-87/003, March 1987.
- USEPA. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW846, Third Edition.
- USEPA, 1988a. Contract Laboratory Program Statement of Work for Inorganic Analysis, Document Number 1LM01.0, March 1988.
- USEPA, 1990. Contract Laboratory Program Statement of Work for Organic Analysis, Document Number 0LM01.0, March 1990 with current revisions.
- USEPA, 1988b. Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses, U.S. EPA Hazardous Site Evaluation Division, February 1, 1988.
- USEPA, 1988c. Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses, U.S. EPA Hazardous Site Evaluation Division, July 1988.

ATTACHMENT A

FIELD SCREENING METHOD SELECTION AND PROCEDURES

MEMORANDUM

DATE: October 31, 1994

TO: Chandler Taylor

FROM: Steve Grumann

RE: Immunoassay Field Screen for NOP Project

An evaluation of four products which were candidates for the immunoassay field screen of TNT for the NOP project has been completed. The four product manufacturers were:

- EnSys, Inc.
- Millipore Corp.
- EM Science (D-Tech)
- Quantix Systems

The criteria used to evaluate the products were: 1) method type (immunoassay or chemical), 2) number of explosives and whether the product can distinguish between explosives compounds, 3) detection limits, 4) procedure, 5) ease of use, 6) equipment needs, 7) correlation to Method 8330, 8) interferences, and 9) cost. The following is a summary of the results of the evaluation and a recommendation of which would be the best product to use for this project.

1. EnSys, Inc. TNT Soil Test Kit

This kit is not immunoassay methodology. It is the same methodology as the field screening done during the RI at NOP. Because this is not an immunoassay kit, this kit was not considered a viable candidate for this project.

2. Millipore Corp. Envirogard TNT Plate Kit

This kit is an immunoassay kit. Literature for this kit indicates that it is qualitative to **semi**-quantitative. Correlation data was not available for this method. Costs for this kit would be approximately \$20 per sample (run once) plus \$500 for the rental of the field lab and spectrophotometer.

3. EM Science DTECH TNT/RDX Test Kit w/ Soil Extraction Pac

This kit is immunoassay methodology and works for soils. The literature for this kit indicates that soil detection limits for RDX and TNT are approximately 0.5 and 0.25, respectively. The literature for the TNT kit indicates it is **semi**-quantitative and states that the kit correlates well with Method 8330 on the **presence or absence** of TNT. Data for RDX indicates that the correlation is good (R=0.9, linear regression slope=1.1) and the kit can be used quantitatively. Because this is the only vendor identified with a RDX kit, it may be valuable to run this kit for

TNT and RDX and try to establish a better correlation for TNT based on site-specific data. The cost of analysis would be approximately \$30 per sample (run once), plus \$300 for the DTECHTOR meter (reflectometer) for reading the results.

4. Quantix Systems TNT Kit

This kit is immunoassay methodology for soil or water analysis. The manufacturer lists the detection limit of the kit at 0.25 ppm in soil. The literature also presents the kit as a quantitative analysis. Correlation between Method 8330 and the kit is good (R=0.96, linear regression slope=0.93). The cost of analysis would be \$21 per sample (run in duplicate), plus we could either: 1) purchase the LabStation for \$5880; 2) lease the LabStation for \$200 to \$325 per month; or 3) participate in the Pilot Project Program with the purchase of a minimum number of kits, and, in addition to receiving training to use the LabStation, return the LabStation after use for no cost.

Based on the evaluation of the products, the Quantix Systems TNT kit and the D-Tech RDX and TNT kits are the recommended products for the immunoassay field screens at NOP. The Quantix kit offers a low cost field screen that has been shown to have a good correlation with Method 8330. The D-Tech kit is more expensive than the others, but can be run for TNT and RDX. Procedures for the Quantix and D-Tech kits are attached.

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QUANTIX TNT IMMUNOASSAY FIELD SCREEN TEST OPERATING PROCEDURES

1.0 GENERAL

QUANTIX TNT is a competitive enzyme immunoassay for the quantitative analysis of TNT in water and soil samples. The basis of the test is the antigen-antibody reaction. TNT conjugate and TNT standards or sample solutions are added to microtiter wells coated with antibodies directed against TNT. Free and enzyme conjugated TNT will compete for the TNT antibody binding sites at the solid phase. Any unbound conjugate is then removed in a washing step. Enzyme substrate and chromagen are added to the wells. Bound enzyme conjugate changes the color of the chromagen, which is measured with a photometer. The absorption is inversely proportional to the TNT concentration in the sample.

2.0 REAGENTS

2.1 REAGENTS AND EQUIPMENT

Each test kit contains sufficient materials for 96 measurements (including standards). Each test kit contains:

- 1 Microtiter plate, (12 strips x 8 wells) 96 wells coated with antibodies to TNT.
- TNT Analytical Standard Solutions, 1.2 ml each:
 0 ppb (negative control), 0.05 ppb, 0.2 ppb, 1.0 ppb, and 20 ppb TNT in water.
- Conjugate, 0.8 ml, red cap.
- Conjugate Diluent, 7 ml, white cap.
- 1 Substrate, 7 ml solution, green cap.
- 1 Chromogen, 7 ml solution, blue cap.
- 1 Stop Reagent (contains 1 M sulfuric acid); 14 ml, yellow cap.
- 4 Empty 4 ml conjugate dilution vials.
- 40 Soil Extraction Bottles, 30 ml plastic bottle with 21 ml acetone.
- 40 Soil Collectors.
- 80 Diluent Tubes with 10.0 ml high purity water.

Quantix MicroReader III Auto Strip Reader.

QuantiPlot Software Cartridge (v3.07).

Plate Shaker, 2 plate size.

Repeating Pipettor.

Adjustable Pipettor, 20-200 ul.

Manual Strip Washer System (vacuum station, flasks, connectors, 8-channel strip washer).

Pipette Tips - 5 boxes, with 96 per box.

Repeater Tips 2.5 ml - 10 pieces per package.

Repeater Tips 5.0 ml - 10 pieces per package.

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2.2 STORAGE COMMENTS

Store the kit at 2-8 C. DO NOT FREEZE.

Return any unused microwells to their original foil bag and reseal them together with the desiccant provided.

The colorless chromogen is light sensitive. Avoid exposing it to direct light.

2.3 INDICATIONS OF INSTABILITY OR DETERIORATION

Any coloration of the chromogen solution is indicative of deterioration and the reagent should be discarded.

A value of less than 0.6 absorbance units for the zero standard may indicate deterioration of reagents.

2.4 WARNINGS AND PRECAUTIONS FOR THE USERS

The stop reagent contains 1 M sulfuric acid. Avoid contact with skin.

Do not use QUANTIX TNT kit past the kit expiration date. Dilution or adulteration of these reagents may result in loss of sensitivity.

Do not interchange individual reagents between kits of differing lot numbers.

Do not use partially used strips.

The kit can be used up to six weeks after opening the package.

3.0 QUANTIX IMMUNOASSAY KIT OPERATION

3.1 PREPARATION OF SOIL SAMPLES

All samples should be stored in the dark and refrigerated until they are analyzed.

The QUANTIX TNT kit provides complete soil sample preparation materials for collecting, extracting, and diluting soil samples prior to immunoassay analysis.

- 1. Eliminate any rocks or gravel from soil, mix thoroughly, and utilize a representative subsample for testing. You may use wet or dried soil.
- Withdraw the plunger of a Soil Collector to the line and pack soil into barrel. Be certain to eliminate air pockets.

- 3. Extrude soil into pre-filled Soil Extraction Bottle with 21 ml of acetone. To speed dispersion, break the soil into small pieces while adding to the acetone.
- 4. Cap the container and shake thoroughly to break up soil. Shake for three minutes by hand. You may prepare and shake several samples at one time.
- Allow soil to settle for several minutes.
- 6. Dilute the acetone extract by transferring 200 μl of extract to 10 ml water in a Diluent Tube using the positive displacement pipette. Dilute again by transferring 200 μl of sample from the Diluent Tube to another Diluent Tube containing 10 ml of water.
- 7. Mix the diluted sample thoroughly prior to analysis. This sample can now be analyzed for TNT concentration in the range of 1.2 ppm to 250 ppm.

3.2 ENZYME IMMUNOASSAY PROCEDURE

3.2.1 Preliminary Comments

- Bring all reagents to room temperature before use.
- Return all reagents to 2 8°C immediately after use.
- 3. Do not allow microwells to dry between steps.
- 4. Reproducibility in any Enzyme Immunoassay (EIA) is largely dependent upon the consistency with which the microwells are washed. Carefully follow the recommended washing sequence as outlined in the EIA test procedure, Section 3.2.5.
- 5. Avoid direct sunlight during all incubations. Covering the microtiter plates is recommended.

3.2.2 Wash Solution Concentrate (10x)

1. This solution is supplied as a 10-fold concentrate and must be diluted 1:10 in distilled or deionized water to prepare the working Wash Solution for all 96 assay wells. Pour the whole bottle of concentrate (100 ml) into a graduated cylinder and dilute to 1.0 liter with distilled or deionized water. The pH of the working Wash Solution should be in the 7.7 to 7.9 range. Prepare small quantities by diluting aliquots of the Wash Buffer Concentrate 1:10 with distilled or deionized water.

3.2.3 Conjugate

1. The TNT enzyme conjugate (enzyme conjugate, bottle with red cap) is provided as a concentrate. Since the diluted enzyme conjugate has a limited stability, only the amount which

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actually is needed should be reconstituted. Before pipetting, the enzyme conjugate should be shaken carefully.

- 2. Prepare the working conjugate solutions as follows:
 - Add 150 µl Conjugate and 1.5 ml Conjugate Diluent to one of the empty conjugate vials provided in the kit. Cap the vial and invert repeatedly to mix the solutions.
 - This quantity of working conjugate is sufficient for three microtiter strips.

3.2.4 Antibody-Coated Microtiter Strips

 Cut the foil pouch open along the transverse side. Remove the number of strips required together with the frame. Return those strips not required and the desiccant pouch to the foil pouch. Fold the pouch closed and seal it with tape. Store at 2 - 8°C.

Note: Partially used strips must not be used again!

Assay Layout

Use the layouts described below when using the Quantix MicroReader.

New Standard Curve

Utilize this Assay Layout when creating a new standard curve.

3.2.5 Test Procedure

1. Insert a sufficient number of microtiter strips into the microwell holder for all standards and sample to be run in duplicate. Record standard and sample positions.

Note: See the recommended layout template above.

- 2. Add 100 µl of standard or prepared sample to separate duplicate wells.
- 3. Add 50 µl of the TNT working Conjugate to each well using a repeating pipette. Mix the strips gently by hand or on a plate shaker.
- 4. Incubate for 30 minutes at room temperature.
- 5. At the end of the incubation period, wash the wells five times with the following procedure: aspirate material from all the wells of column 1 using the microwell washer or other wash method. Then fill all the wells of column 1 with working Wash Solution. Complete this sequence for each successive column of wells until all columns of wells are filled with Wash Buffer Solution.

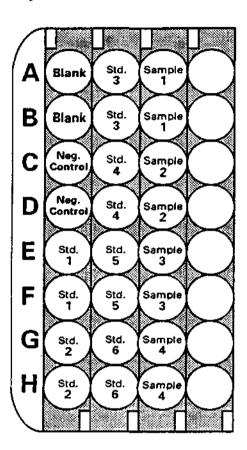
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Assay Layout

Use the layouts described below when using the Quantix MicroReader.

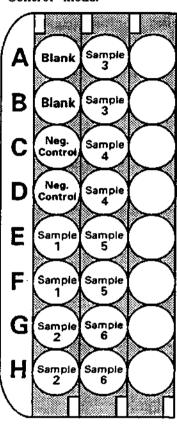
1. New Standard Curve.

Utilize this assay layout when using the "Update Curve" mode.



2. Stored Standard Curve.

Use this Assay Layout when using a stored standard curve and the "Blank and Negative Control" mode.



Repeat the whole aspiration/fill sequence four more times.

Finally, tap the plate upside down on several layers of absorbent tissue to remove residual droplets of wash solution.

Note: When inverting the plate, be sure to squeeze the plastic frame at the center of the long edges to prevent the strips from falling out of the frame.

- 6. Add 50 μl of Substrate to each well with a repeating pipette. Immediately add 50 μl of Chromogen to each well. Mix thoroughly by hand or on a plate shaker and incubate for 30 minutes at room temperature in the dark.
- Add 100 μl of Stop Solution to each well. Mix well.
- 8. The absorbance must be measured at 450 mm against an air blank within 60 minutes of the stop solution addition. Instructions for absorbance measurement are given in Section 4.0.

4.0 ABSORBANCE MEASUREMENT AND CONVERSION TO CONCENTRATION VALUES

The optical density of the solutions in the microcups will be determined with a Quantix MicroReader.

Sample concentrations will be determined with a standard curve developed along with the samples. Microcups A1 and B1 must always be used as blanks, and Microcups C1 and D1 must contain negative control. Standards are tested in duplicate to fill the remaining microcups of the two strips when a new standard curve is being developed.

When the reader has warmed up, the screen will display:

Run a Test? Press the Yes key

Then follow the sequence in the table below.

Screen Prompt	RESPONSE - Press the indicated keys	
Pick test to run	test number 11 ENTER	
Run [Test Name] Test?	YES	
Curve stds in strip? [Y] or just BLK and NC? [N]	YES	
Read how many wells? [16-16]	16 ENTER	
Please insert strip Press [Y] to continue	YES	
The reader will now read wells 1-16 of the assay the R ₀ term and negative control microcups C1 calculation. The reader will print out the absorb point and a graph of the standard curve.	and D1 as the R _m term of the concentration	
Store the curve [Y]? or abort the test [N]?		
If the curve gave acceptable results, press Yes to store the new standard curve in memory.	YES	
Continue test?	Yes to read samples.	

D TECH TNT/RDX DETECTION SYSTEM OPERATING PROCEDURES

1.0 GENERAL

The D TECH system uses an organic solvent to extract TNT and RDX compounds from soil for semi-quantitative analysis. Following extraction, immunoassay technology is used to analyze for trace amounts of TNT and RDX. Antibodies for TNT, RDX, and closely related compounds are linked to solid particles. These antibodies are mixed with the extracted sample and then collected on the membrane of a cup assembly. A color developing solution added to the surface of the cup assembly develops a color inversely proportional to the concentration of TNT and RDX equivalents in the sample. This color is then compared with a color standard to determine the TNT and RDX concentration.

2.0 REAGENTS

2.1 TNT/RDX SOIL EXTRACTION PAC

All items found in the extraction pac are enclosed in a special kit. At the conclusion of the test, the components can be placed in the package for proper disposal.

- 4 Bottle 1
- 4 Bottle 2
- 4 Soil Sampling Tubes
- 4 Pipette Tips
- 4 Red Dot Labels (to indicate that a Bottle 2 has been used)
- 1 Instruction Guide
- 1 Used Kit Label

2.2 TNT/RDX EXPLOSIVES TEST KITS

- 4 Bottle A
- 1 each Reagent C,D,E,F
- 4 TNT/RDX Vials
- 4 Filter Tips
- 4 TNT/RDX Reference
- 4 White Cup Assembly
- 4 Calibrated Pipettes
- 4 Red Dot Labels (to indicate used Bottle A components)
- 4 Data labels for cup assembly
- Color Card
- 1 Instruction Guide
- 1 Used Kit Label

2.3 ADDITIONAL ACCESSORIES

Timing Device (minutes)
DTECHTOR Meter

2.4 STORAGE COMMENTS

All materials in these kits have excellent stability at room temperature and under refrigeration. Expiration dating is provided on the package label.

2.5 HEALTH AND SAFETY

MSDSs are supplied with purchase of equipment and should be read before use of test.

Protect eyes with safety glasses Protect skin with protective gloves

3.0 SOIL EXTRACTION PROCEDURE

3.1 SAMPLING

- 1. Break up the soil and mix as necessary so that it is uniform and free of rocks, wood, leaves, and other debris. Draw back the soil sampling tube plunger until it stops. Push the soil sampling tube into the soil several times with a twisting action to firmly pack and fill the tube. Sandy soil may require a scooping action to fill the tube. Remove excess soil from the external surface of the sampling tube and barrel end.
- 2. Dispense the soil into Bottle 1 by positioning the barrel into the neck of the bottle and firmly pushing the plunger. Squeezing the barrel of the soil sampling tube will help to expel a tightly packed sample. If soil lodges in the neck of the bottle, use the sampling tube to push it into the bottle. If soil adheres to the threads of the bottle neck and cap, wipe clean before placing cap on the bottle. Cap bottle tightly.

3.2 EXTRACTION FROM SOIL

- 3. Mix the soil and liquid in Bottle 1 by shaking continuously for at least 3 minutes.
- 4. Allow the soil to settle until a clear liquid layer forms. Some soils will settle more slowly than others. Soils with clay may require 5-10 minutes to settle sufficiently.

3.3 DILUTING THE EXTRACTION SOLUTION

- 5. Remove the cap from Bottle 2
- 6. Place an unused tip on the pipette

- 7. Fully depress the plunger of the pipette. With the plunger fully depressed, place the pipette tip into the clear liquid layer and slowly release the plunger. Take care not to aspirate any soil into the pipette tip.
- 8. Dispense the contents of the pipette tip into Bottle 2 by placing the pipette tip into the liquid and depressing the plunger. Mix Bottle 2 thoroughly. Replace the cap tightly on Bottle 1 and return it to the tray. Place the used pipette tip in the right side tray compartment.
- 9. Use the contents of Bottle 2 as the sample in the TNT and RDX tests (Section 4.0). Following sample removal from Bottle 2, cap Bottle 2 tightly, attach a red dot label, and return to the tray. After the last extraction has been performed, place the "Used Kit" label on the Soil Extraction Pac box to seal it shut.

4.0 TNT/RDX EXPLOSIVES TEST KIT PROCEDURE

The TNT and RDX test kits have the same operating procedures, except for the different time period in step 4, as noted below.

- 1. Using a new calibrated pipette, transfer 1 ml of Bottle 2 solution to Bottle A. Snap the filter tip on Bottle A. Gently mix. Re-cap Bottle 2 as discussed in Section 3.3 above.
- 2. Squeeze Bottle A to fill the TNT/RDX vial to a level between the two lines (approximately 13-14 drops). Gently mix.
- 3. Squeeze the contents of Reagent C (white cap) to fill the TNT/RDX Reference vial to a level between the 2 lines. Gently mix.
- 4. Allow the solutions in the vials to stand for 2(TNT)/5(RDX) minutes after dispensing the liquid. The solutions in the vials will remain hazy. After 2/5 minutes, pour the contents of the TNT vial onto the T (test) side of the cup assembly. Pour the contents of the Reference vial onto the R side of the cup assembly. Allow liquid to drain completely through on both sides.
- 5. Add approximately 8-12 drops of Reagent D solution (yellow cap) into each side of the cup assembly. Drain completely.
- 6. Add approximately 5 drops of Reagent E solution (blue cap) to each side of the cup assembly. Be sure to add this solution immediately to the second well after addition to the first well. Drain Completely.
- 7. Read results when color of R(left) side of cup assembly matches the color of the reference bar of the Color Card or the DTECHTOR meter indicates the correct color has been reached. The color development time is approximately 10 minutes at 70F. More time is required at lower temperatures and less time is required at higher temperatures.

Note: To preserve the color for up to 4 hours (optional), add approximately 8 drops of Reagent F solution (red cap) into each side of the cup assembly. Drain completely.

5.0 DTECHTOR METER SET UP

The DETECHTOR light sources must be calibrated whenever the meter is turned on. Calibrators are provided with the meter for this purpose. The Calibrator must be clean and white to ensure valid results.

- Insert Calibrator into the Meter Head and hold firmly in place.
 (ZERO)
- Press the Square Button 1 time. When calibration is complete the meter will display.....
 (SET)
- 3. Remove Calibrator and return it to its protective canister. Display remains.... (SET)
- Press the Square Button 1 or 2 times to select meter program #1(TNT) or #2(RDX). (SET#1/SET#2)
- 5. Insert Cup Assembly (test) into the Meter Head and firmly hold in place. (TEST#1/TEST#2)
- 6. Press the Square Button 1 time. (---) then (SET#1/SET#2)

Note: If the meter displays "WAIT", remove Cup Assembly. Allow reference color to develop further and try again.

7. Remove the Cup Assembly (test). The meter will display the reflectance units of the reference side of the Cup Assembly along with #1 in the upper right corner of the display window.

The lower the reflectance unit number displayed, the darker the color. The longer the color is allowed to develop, the lower the reference reflectance unit number will be. The target reference reflectance for the test is 230 (TNT)/330 (RDX). Therefore, if the reference reflectance is greater than the target, the color development time is needed. If the reference reflectance is less than the target, the color development has been allowed to proceed too long. The greatest accuracy in quantitating the test result with the meter is to read the color development when the reference reflectance is as close to the target number as possible.

8. Press the Square Button then the slide (on/off) switch. The meter will display the reflectance units of the test side of the Cup Assembly along with #2 in the upper right corner of the display window.

9. Press the Square Button then the slide (on/off) switch again. The meter will display the percent relative reflectance (the meter reading obtained when the DTECHTOR is in the regular mode) along with #3 in the upper right corner of the display window.

For example:

Use the DETECHTOR Table and the meter reading to determine the concentration of TNT/RDX.

- 10. Record the result then press the Square Button while holding the Cup Assembly in place. 11----)
- 11. Key in a 4 digit label. (optional)
- 12. Remove Cup Assembly (SET#1/SET#2)
- 13. Insert next Cup Assembly (test) and repeat steps 5-12.

THE DTECHTOR TABLE FOR SOIL SAMPLES

The DTECHTOR Reading	TNT Equivalents (ppm)	The DTECHTOR Reading	RDX Equivalents (ppm)
LO	<0.5	LO	<0.5
1 - 15	0.5 - 1.5	1 - 20	0.5 - 1.5
15 - 45	1.5 - 3.0	20 - 45	1.5 - 2.5
45 - 60	3.0 - 4.0	45 - 60	2.5 - 4.5
60 - 75	4.0 - 5.0	60 - 80	4.6 - 6.0
Ш	>5.0	Щ	>6.0

D-MEADWCDAPINDTECHTAB.

November 1994

B07NE003701-05457

ATTACHMENT B

USACE MRD LETTER OF VALIDATION FOR HUNTINGDON/TCT-ST. LOUIS

01/10/94 10:40 MANTE THO TALL 101 01 01 01 01



DEPARTMENT OF THE ARMY MISSOURI RIVER DIVISION, CORPS OF ENGINEERS P.O. BOX 103, DOWNTOWN STATION OMAHA NEBRASKA 68101-0103



REPLY TO

August 5, 1993

Environmental, Hazardous, Toxic and Radioactive Waste Division

Twin City Testing Corporation 1908 Innerbelt Business Center Drive St. Louis, Missouri 63114

Gentlemen:

This correspondence addresses the recent revalidation of Twin City Testing Corporation, by the U.S. Army Corps of Engineers (USACE) for hazardous, toxic and radioactive waste analysis.

The laboratory has successfully analyzed audit samples as listed below:

<u>METHOD</u>	PARAMETERS	MATRIX
8240	Volatile Organics	water
		water
8010	Halogenated Volatile Organics	·· -
8020	Aromatic Volatile Organics	water
8270	Semivolatile Organics	water
8270	Semivolatile Organics	sediment
8080	Organochlorine Pesticides	water
8080	Polychlorinated Biphenyls	water
8080	Polychlorinated Biphenyls	sediment
\$080	toflemfortwater primaris	5552115115
SW-846	TAL Metals	water
SW-846	TAL Metals	sediment
418.1	TRPH	water
418.1	TRPH	scil
420.2		
8150	Herbicides	water
9010	Cyanide	water
9060	Total Organic Carbons	water
5000	100d1 019 010 010 010 010 010 010 010 010 01	
300.0	Anions	water
2222	Emlegine	water
8330	Explosives	soil
8330	Explosives	2011

Remarks: TAL Metals: 23 EPA Contract Laboratory Program, Target Analyte List (TAL) metals (aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc.)

Based on the successful analysis of the audit samples indicated in the table in paragraph two, your laboratory is revalidated for multimedia sample analysis by the above methods. A full validation of eighteen (18) months was approved by the USACE Contract Laboratory Evaluation Committee on July 28, 1993.

The expiration date of validation is February 3, 1995. The Chemistry Branch of the Hazardous, Toxic and Radioactive Waste Mandatory Center of Expertise may schedule and conduct an on-site audit at any time during the 18-month validation period to evaluate lab performance if deemed necessary. USACE reserves the right to conduct laboratory audits or to suspend validation status for any or all of the listed parameters if deemed necessary. It should be noted that your laboratory may not subcontract USACE analytical work to any other laboratory location without the approval of this office. This laboratory validation does not guarantee the delivery of any analytical samples from a USACE Contracting Officer Representative.

If you have any questions or comments, please contact Ms. Paulette Lewis at (402) 221-7494.

Sincerely,

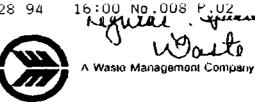
Marcia C. Davies

Chief, Environmental, HTRW Division HTRW and Engineering Directorate

ATTACHMENT C

DOUGLAS COUNTY LANDFILL SPECIAL WASTE ANALYTICAL REQUIREMENTS

Douglas County Landfill 14320 No. 216th St. Bennington, Nebraska 68007 1-402/478-5141



Douglas County Landfill/ A Division of Waste Management Special Waste Analytical Requirements May 12, 1993 Page 1 of 2

Required Y/K	EPA HW Number	Parameter	Regulatory Level	See Note
_	D001	Ignitability.	> 140 Fahren	1
			2 - 12.5 <	2
		•	non-reactive	3
\overline{V}		Arsenic-	5.0 ppm	4
		Barium	100.0 ppm	4
		Cadmium.	1.0 ppm	4
		Chromium	5.0 թթա	4
		Lead.	5.0 ppm	4
1		Mercury.	0.2 ppm	4
ーレー		Selenium	1.0 ррв	4
~		Silver	5.0 ppm	4
		Endrin	0.02 ppm	5
		Lindane	0.4 ppm	5
	D014	Methoxychlor	10.0 ppm	5
		Toxaphene	0.5 ppm	5
		2,4-D	10.0 ppm	5
		2,4,5 - TP Silvex	1.0 ppm	5
<u> </u>		Benzene-	0.5 ppm	6
	D019	Carbon Tetrachloride-	0.5 ppm	6
	D020	Chlordane	0.03 թթա	
_ ~	D021	Chlorobenzene:	100.0 ррш	6
- 1	D022	Chloroform-	6.0 ppm	6
- نسسة	D023	o - Cresol	200.0 ppm	6,
<u></u>	D024	m - Cresol	200.0 բթա	6,
	D025	p - Cresol	200.0 ррв	6,
1		Cresol-	200.0 ppm	6,
استآسا	D027	1,4 - Dichlorobenzene-	7.5 ppm	6
1.		1,2 - Dichloroethane-	0.5 ppm	6
		1,1 - Dichloroethylene-	0.7 ppm	6
سسا	D030	2,4 - Dinitrotoluene -	0.13 ppm	6,
		Heptachlor and its Hydroxide	Ф.008 ррж	
- W		Hexachlorobenzene -	0.13 թթա	6,
ニレ	D033	liexachloro - 1,3 - butadiene	- 0.5 ppm	6
سا	D034	liexachloroethane-	3.0 ppm	6
سيا	D035	Methyl Ethyl Ketone-	200.0 ррв	6
	D036	Nitrobenzene -	2.0 ppm	6
<u></u>		Pentachlorophenol ~	100.0 թբա	6
	0038	Pyridiner	5.0 ppm	6,
		Tetrachloroethylene/	0.7 ррш	6
		trichloroethylene -	0.5 թթա	6
		2,4,5 - Trichlorophenol-	400.0 թթա	6
		2,4,6 - Trichlorophenol-	2.0 ppm	6
<u></u>	D043	Vinyl Chloride -	0.2 ppm	8

a division of Waste Management of Nebraska, Inc.



Douglas County Landfill Special Waste Analytical Requirements May 12, 1993 Page 2 of 2

Required Y/N	DCLF Number	Parameter	Regulatory Level	See Note
	W001	Paint Filter	Must Pass	-
	W002	Total Solids	40% Solide	
	W003	Ash Content	Non Specific	9
سرا	W004	Total Phenolics	1% Limit	
	W005	Cyanide	10.0 ppm	10
شرا	W006	Sulfide	10.0 ppm	10
	W007	PCB's	25.0 ppm	9,11
	800W	Solvents	100.0 ppm	9,12
<u>سن</u>	K008	Copper	20.0 ppm	4
	W010	Nickel	20.0 ppm	4
	W011	Zinc.	50.0 ppm	4

Notes

- 1 Modified Clevelend Open Cup. Results must be an exact temperature.
- 2 If pH = 10 or more, must test for alkalinity, if pH = 4 or less, must test for acidity.
- 3 See 40 CFR Chapter I Section 281.23, Characteristic of Reactivity.
- 4 Totals may be tested instead of TCLP concentrations. If totals results exceed TCLP limits, then TCLP must also be tested, for specific metals.
- 5 May be required, depending on likelihood of presence or unknown origin of waste.
- 6 Required by TCLP.
- Quantitative limit is greater than the calculated regulatory level. The quantitative limit, therefore, becomes the regulatory level.
- 8 If o,m, and p Cresol concentrations cannot be differentiated, the total Cresol (D026) concentration is used.
- 9 May be required on a case by case basis.
- 10 If results exceed 10 ppm, must test for total releasable.
- If results exceed 25 ppm, must test for specific aroclors.
- 12 Method 8240, SW846, 29 listed solvents, if any single parameter exceeds 100 ppm, must use mass spec confirmation for identification.